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RESEARCH AND DEVELOPMENT PROGRAM INTRINSIC RELIABILITY SUBMINIATURE CERAMIC CAPACITORS

SECOND QUARTERLY PROGRESS REPORT

PERIOD: 1 SEPTEMBER 1962 - 30 NOVEMBER 1962

TO

U. S. ARMY SIGNAL RESEARCH & DEVELOPMENT LABORATORY FORT MONMOUTH, NEW JERSEY

CONTRACT NO. DA-36-039-SC-90705 D. A. PROJECT NO. 3A99-15-001

SPRAGUE ELECTRIC COMPANY NORTH ADAMS, MASSACHUSETTS

NO 015

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RESEARCH AND DEVELOPMENT PROGRAM

INTRINSIC RELIABILITY

SUBMINIATURE CERAMIC CAPACITORS

Second Quarterly Progress Report

Period: 1 September 1962 - 30 November 1962

Object of Study: To conduct investigations leading to the approaches for the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

Contract No. DA-36-039-SC-90705 D. A. Project No. 3A99-15-001

Controlling Specifications:

Signal Corps Technical Guidelines, "Reliability Long Life Component Studies," 3 November 1961 Signal Corps Technical Requirements No. SCL-2101N, 14 July 1961

Report Prepared by:

T. I. Prokopowicz

F. B. Schoenfeld

J. H. D. Folster

TABLE OF CONTENTS

			Page
SECTION 1	-	PURPOSE	1 -1
SECTION 2	-	ABSTRACT	2-1
SECTION 3	-	PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES	3-1
SECTION 4	~	FACTUAL DATA 4.1 Pre-Matrix Testing 4.2 Test Results 4.3 Study of Charge and Discharge Currents 4.4 Matrix I Testing	4-1 4-2 4-4 4-6
SECTION 5	-	CONCLUSIONS	5-1
SECTION 6	-	PROGRAM FOR NEXT QUARTER	6-1
SECTION 7	-	IDENTIFICATION OF PERSONNEL	7-1
SECTION 8	_	DISTRIBUTION LIST	8-1

PURPOSE

The purpose of this contract is to carry out research work for a period of 18 months commencing June 1, 1962, and ending November 30, 1963, involving investigations leading to approaches to the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

In particular, this involves the following:

- (1) Establishment of Matrix I test conditions through a series of pre-matrix tests.
- (2) Development and evaluation of a short-term test to eliminate early failures effectively without shortening the time to the wearout mode of failure.
- (3) A determination of the failure rate as a function of voltage and temperature through Matrix I and Matrix II testing. From the data thus obtained, derating curves will be derived and overall failure rates for operating conditions will be estimated.
- (4) Compilation of quarterly progress reports in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.
- (5) Compilation of a final report in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.

ABSTRACT

A detailed discussion on the results of pre-Matrix I testing is presented in this report. Data and graphs are included. This pre-matrix work has been centered about an effort to predict the life of a capacitor by measuring its discharge current. Among the relationships examined in the course of this study are those between:

(1) resistance and temperature; (2) initial resistance and time to failure; and

(4) intermediate resistance and time to failure. The study is continuing.

PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

- (1) The First Quarterly Progress Report, covering the period 1 June 1962 31 August 1962, was submitted for U. S. Army Signal Supply Agency approval during this quarter. Approval was received, and the report was distributed per USASSA instructions.
- (2) Miss Jeanne Allen, U. S. Army Electronics Research and Development Agency, visited the North Adams plant of the Sprague Electric Company on November 27, 1962. The purpose of this visit was to review progress on this program.

FACTUAL DATA

4.1 Pre-Matrix Testing

The purpose of pre-Matrix I testing is to develop and evaluate short-term tests for the elimination of early failures. The most promising of these tests will then be statistically evaluated in matrix studies involving large numbers of units.

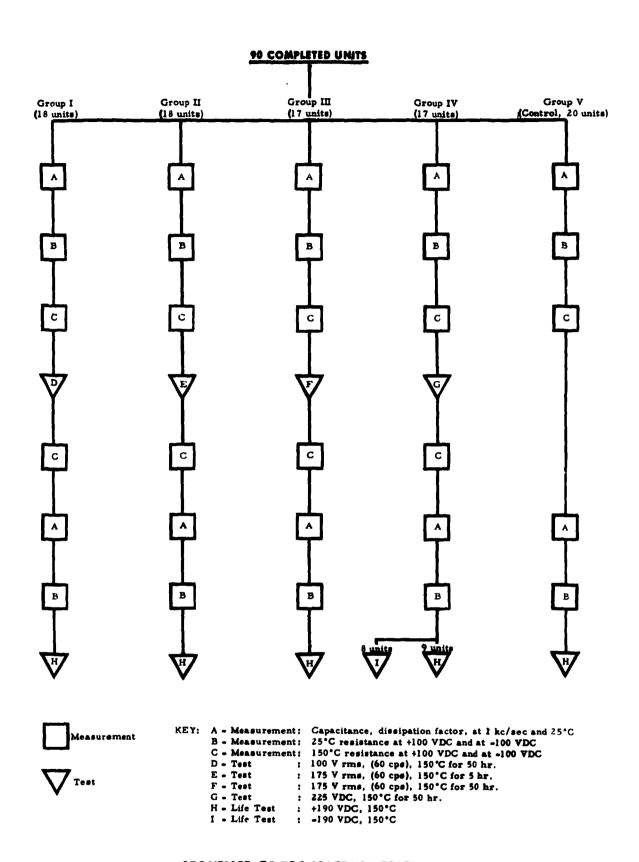
As reported in the First Quarterly Progress Report, 90 units of 4000 $\mu\mu$ f capacity were chosen for the initial pre-matrix testing experiments.

The ceramic material in these units was the C67 formulation, which has a dielectric constant of 2000. The dielectric thickness of the units was 0.0025 in. Each capacitor was enclosed in a molded cylindrical case of 0.095 in. diameter and 0.250 in. length.

The units were divided into five groups, each group undergoing a series of tests and measurements as described in Figure 1 of this report. As can be seen in this figure, this pre-matrix testing included measurements of the electrical resistance of the units at various stages of the test sequence before the life test.

Measurements were made on all five test groups for resistance at 25°C and 150°C with voltages of +100 VDC, -100 VDC applied. In addition, resistance was periodically measured on the units undergoing test at +225 VDC, 150°C, for 50 hr (Test Group IV).

While in this report the voltage conditions for the various tests are given in terms of positive and negative, it is not intended to imply that the test units were polar when manufactured. A positive and negative terminal was arbitrarily assigned to the units because the units were to undergo several DC measurements and tests.



SEQUENCE OF PRE-MATRIX I TESTING

FIGURE 1

Periodic resistance measurements were made during the accelerated life test to which all units were subjected. These measurements were made without interruption of the test. This was accomplished by measuring the voltage drop across each unit and also across a 1 $M\Omega$ resistor permanently in series with it. These voltage drops were then used to calculate the unit's resistance.

4.2 Test Results

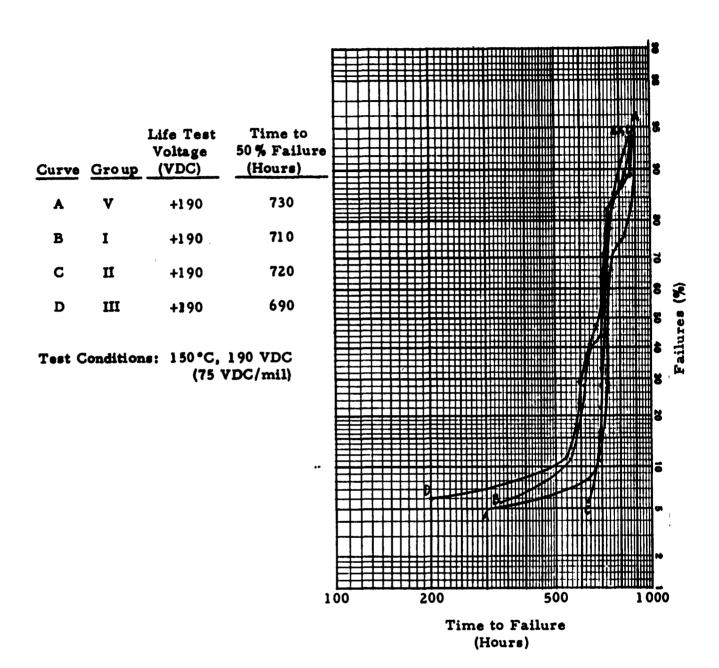
There were no electrical resistance failures before life testing. At Points B and C in Figure 1, the resistance remained above the defined limits. As indicated in subsequent data, the choice of failure definition was not entirely arbitrary, particularly for the 150°C condition. As the data reveal, a resistance of 100 M Ω or less at 150°C is a decrease of approximately three orders of magnitude from a new, unstressed unit. A resistance change of this degree definitely indicates that the capacitor is wearing out.

Figures 2 and 3 show a comparison of the life test performances of the five groups of units. In examining these data, it must be borne in mind that Life Tests H and I shown in Figure 1 are at severe conditions, i.e., 150°C, approximately 75 V/0.001 in. These conditions were chosen in order to obtain useful life performance data in a reasonable period of time.

A comparison of the life test performances of Groups I, II, and III with Group V, the control group, indicates that the AC burn-in voltages applied to these groups (Tests D, E, F in Figure 1) before life test had little or no effect on the average time to failure.

The application of +225 VDC for 50 hr (Test G in Figure 1) produced a polarity on the Group IV units which resulted in a wide variability of life times on the life test. As could be expected, the life performance of the Group IV units subjected to the +225 VDC burn-in and tested at +190 VDC were inferior to those of the control group (Group V). The life performances of the Group IV units tested at -190 VDC were superior to those of the control units. This difference in performances is due to the predominantly ionic nature of titanate dielectric wearout. This ionic nature may explain why AC burn-in had negligible effect on the life performance of the units, particularly since the temperature rise of a unit subjected to 60 cps AC voltage is calculated to be approximately 2°C above ambient.

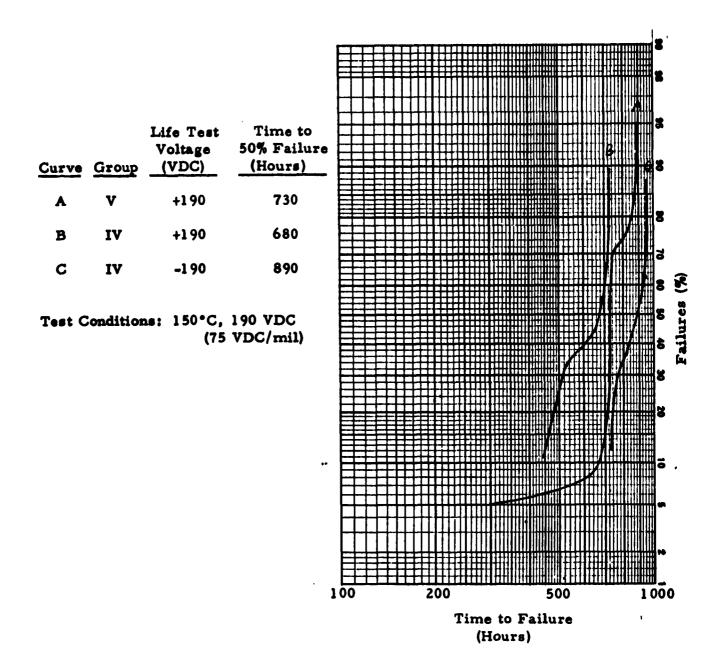
¹ Linden Laboratories, Inc., "High Temperature Ceramic Dielectrics," Report No. 12, October 15, 1961, Contract No. DA-36-039-SC-78912.



NUMBER OF FAILURES VS TIME TO FAILURE

(Definition of Failure: electrical resistance <100 $M\Omega$ at test conditions)

Figure 2



NUMBER OF FAILURES VS TIME TO FAILURE

(Definition of Failure: electrical resistance <100 $M\Omega$ at test conditions)

Figure 3

Figure 4 shows the relationship between resistance and temperature for several units. The units used in this comparison were those which were life-tested at +190 VDC, $150\,^{\circ}$ C. At the beginning of the life test, these units had a resistance of approximately $100,000~M\Omega$, but at its end, the resistance had dropped to about $20~M\Omega$. At this point, the temperature in the life test oven was reduced to $25\,^{\circ}$ C while the life test voltage was maintained. The resistance of the units was measured at several intermediate temperatures. It is notable that the activation energy for conduction of degraded titanate dielectrics is 0.79~eV, a figure which is close to that for nondegraded units. 2

An attempt was made to relate the resistance of units before life test with time to failure on life test. The data obtained from this study are presented in Figures 5-10. The resistance measurement was made after 30 min of electrification with 100 VDC at 150°C. The history of the units undergoing this test is described in Figure 1. The data indicate that there is no useable relationship existing between resistance before life test and time to failure.

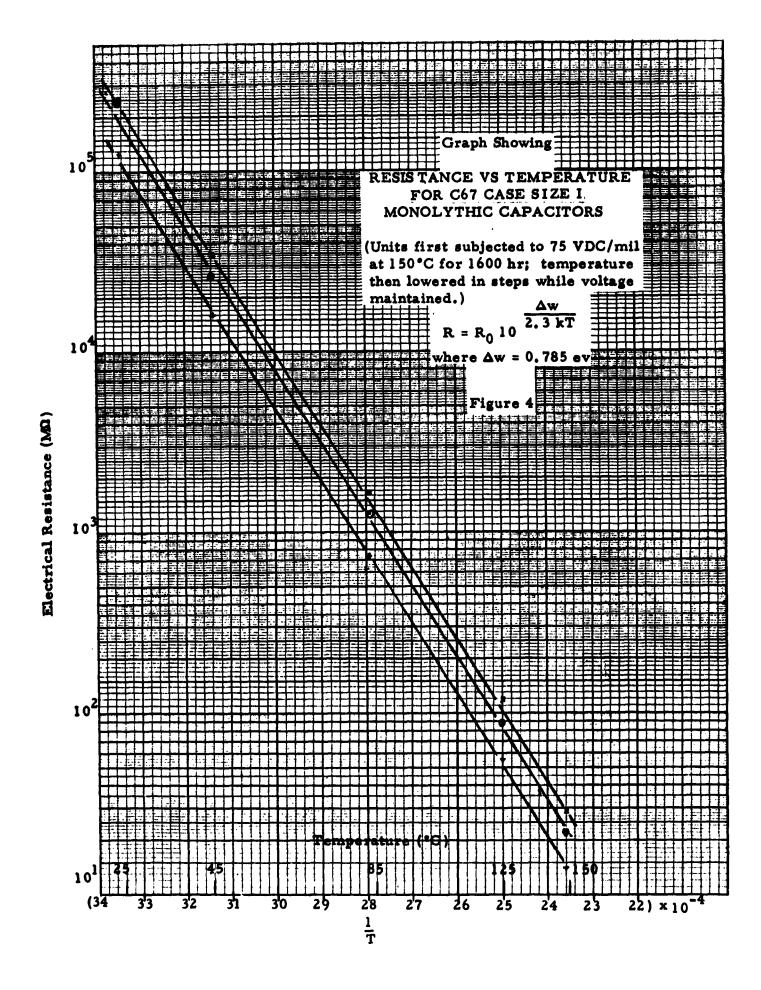
Figures 11-16 describe an attempt to relate resistance symmetry before life test with time to failure on life test. The history of the units used in this study is presented in Figure 1. The ratio of R+/R- is derived from measurements of resistance at +100 VDC and -100 VDC after approximately 30 min of electrification at 150°C. The data indicate that there is no relationship between resistance symmetry and time to failure.

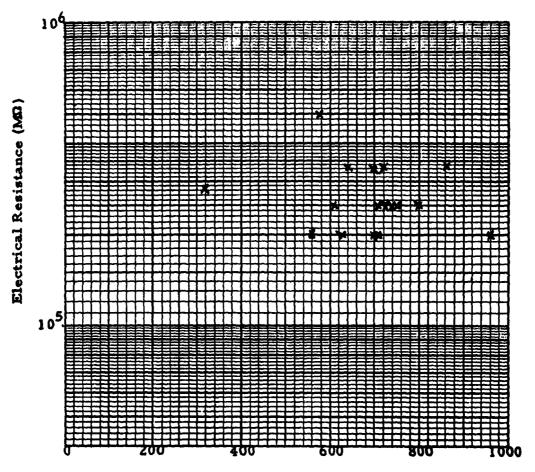
It is significant that the Group IV units in general, when compared to the other groups, show virtually no symmetry with respect to the ratio R+/R-. This behavior is similar to the experience of Kunin and Chikin³ with titania ceramics. This general lack of symmetry indicates that some degradation or wearing occurred during Test G.

Figures 17-22 describe an attempt to relate time to failure on life test with resistance of the test capacitors after 100 hr of life test. It can be seen on comparing these figures with Figures 5-10 that after 100 hr

²Brown, F., "Dielectric Absorption in Ferroelectric Titanates," <u>American</u> Ceramic Society Journal, 42:350-4, (1959).

³Kunin, V., and Chikin, A., "Change of the Dielectric Properties of Rutile Ceramics During the Passage of Current and During Heating," Soviet Physics Solid State, 2:2101-6 (1961).





Time to Failure (Hours)

Testing Sequence

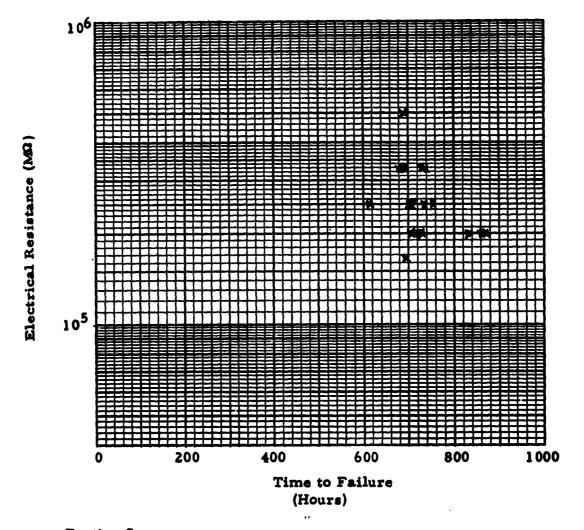
Test D

Measurement C

Test H

RELATIONSHIP BETWEEN
MEASUREMENT C
AND
TIME TO FAILURE ON TEST H
FOR GROUP I UNITS

(Definition of Failure: electrical resistance < 100 $M\Omega$ at Test H conditions)



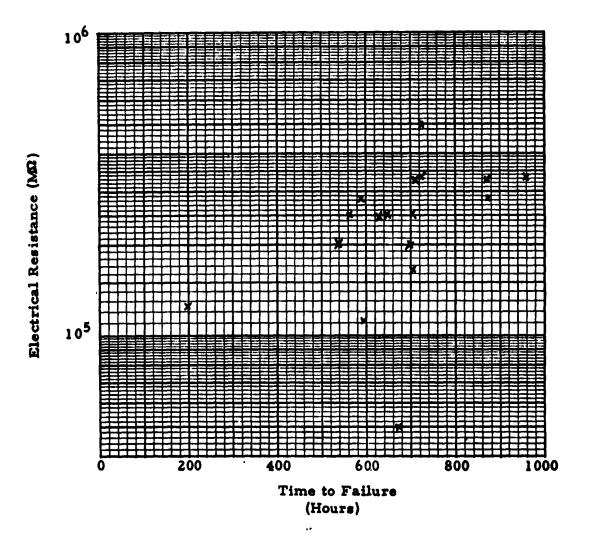
Test E

Measurement C

Test H

RELATIONSHIP BETWEEN MEASUREMENT C AND TIME TO FAILURE ON TEST H FOR GROUP II UNITS

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)



Test F

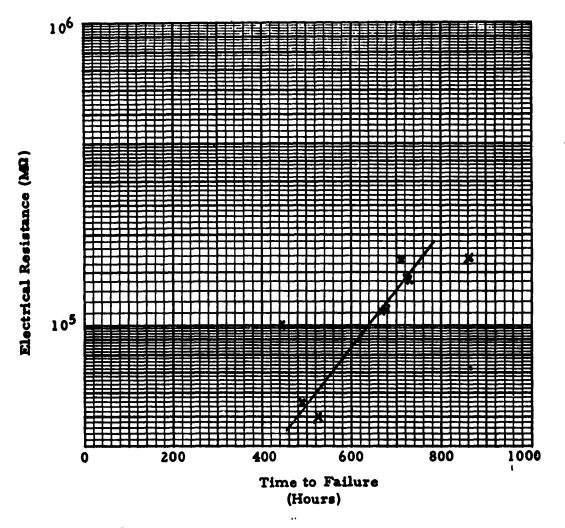
Measurement C

Test H

RELATIONSHIP BETWEEN MEASUREMENT C AND TIME TO FAILURE ON TEST H FOR GROUP III UNITS

(Definition of Failure: electrical resistance <100 $M\Omega$ at Test H conditions)

Figure 7



Test G

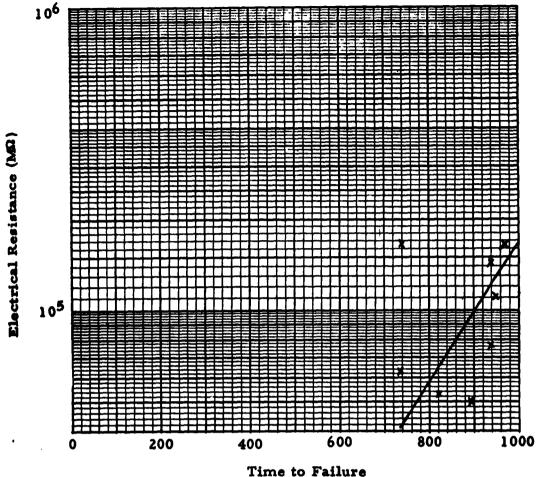
Measurement C

Test H

RELATIONSHIP BETWEEN MEASUREMENT C AND TIME TO FAILURE ON TEST H FOR GROUP IV UNITS

(Definition of Failure: electrical resistance $< 100 \text{ M}\Omega$ at Test H conditions)

Figure 8



(Hours)

Testing Sequence

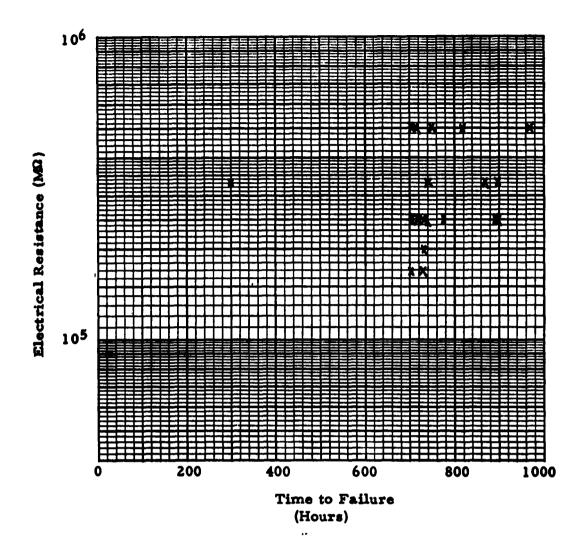
Test G

Measurement C

Test I

RELATIONSHIP BETWEEN
MEASUREMENT C
AND
TIME TO FAILURE ON TEST I
FOR GROUP IV UNITS

(Definition of Failure: electrical resistance < 100 M Ω at Test I conditions)



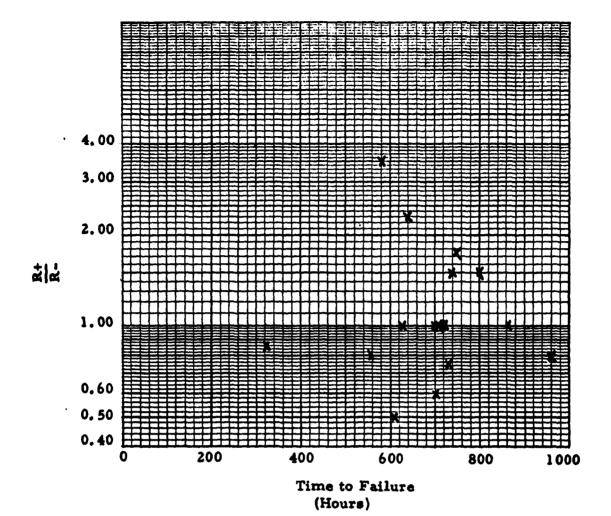
Measurement C

Test H

RELATIONSHIP BETWEEN
MEASUREMENT C
AND
TIME TO FAILURE ON TEST H
FOR GROUP V UNITS

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)

Figure 10



Test D

Measurement C (+100 VDC, 30 min)

Measurement C (-100 VDC, 30 min)

Test H

RELATIONSHIP BETWEEN

RDERIVED FROM MEASUREMENT C

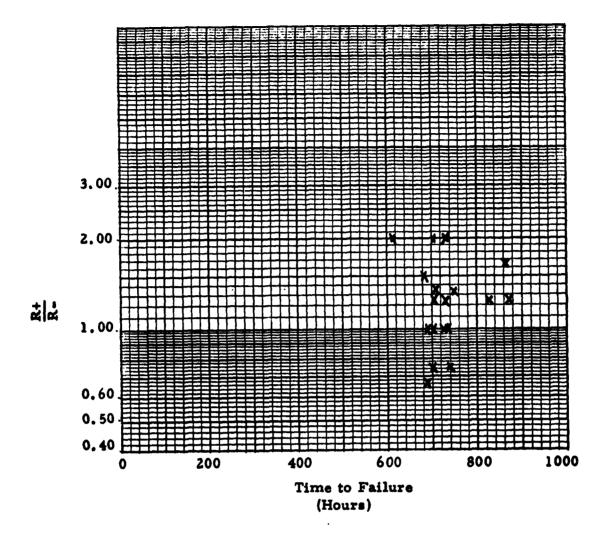
AND

TIME TO FAILURE ON TEST H

FOR GROUP I UNITS

(Definition of Failure: electrical resistance <100 $M\Omega$ at Test H conditions)

Figure 11



Test E

Measurement C (+100 VDC, 30 min)

Measurement C (-100 VDC, 30 min)

Test H

RELATIONSHIP BETWEEN

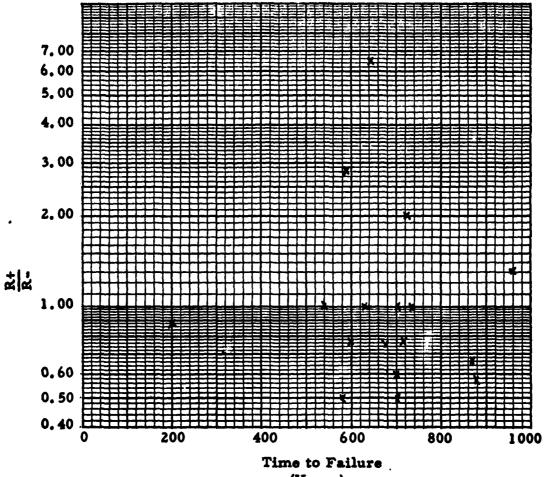
R+ DERIVED FROM MEASUREMENT C

AND

TIME TO FAILURE ON TEST H

FOR GROUP II UNITS

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)

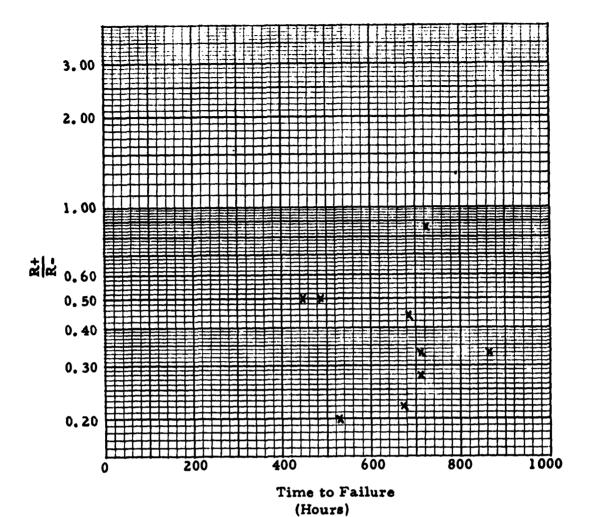


(Hours)

Measurement C (+100 VDC, 30 min) Measurement C (-100 VDC, 30 min) Test H

> RELATIONSHIP BETWEEN DERIVED FROM MEASUREMENT C AND TIME TO FAILURE ON TEST H FOR GROUP III UNITS

(Definition of Failure: electrical resistance $<100 \text{ M}\ \Omega$ at Test H conditions)



Test G

Measurement C (+100 VDC, 30 min)

Measurement C (-100 VDC, 30 min)

Test H

RELATIONSHIP BETWEEN

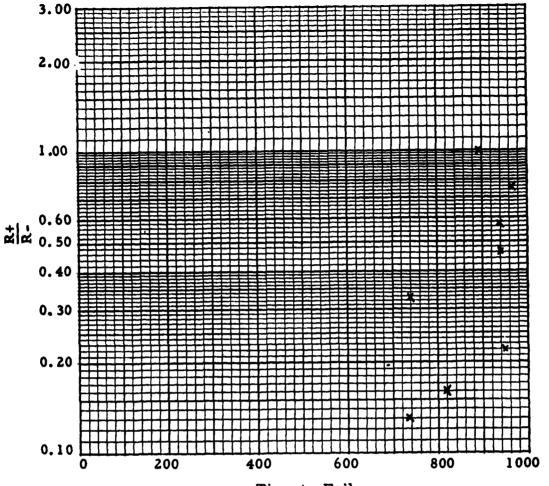
R+ DERIVED FROM MEASUREMENT C

AND

TIME TO FAILURE ON TEST H

FOR GROUP IV UNITS

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)



Time to Failure (Hours)

Test G

Measurement C (+100 VDC, 30 min)

Measurement C (-100 VDC, 30 min)

Test I

RELATIONSHIP BETWEEN

R+
DERIVED FROM MEASUREMENT C

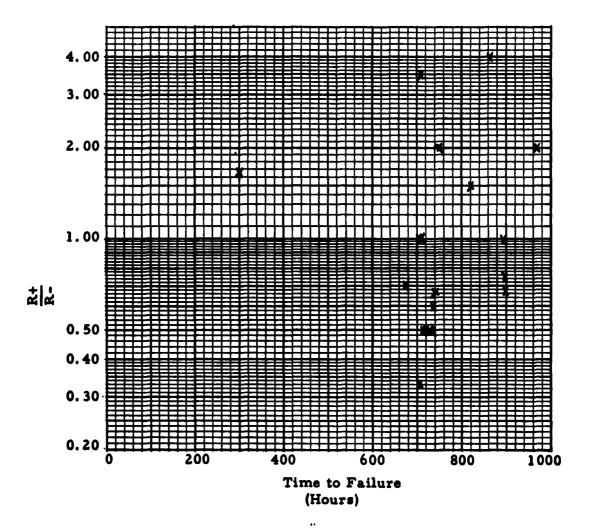
AND

TIME TO FAILURE ON TEST I

FOR GROUP IV UNITS

(Definition of Failure: electrical resistance <100 MΩ at Test I conditions)

Figure 15



Measurement C (+100 VDC, 30 min)
Measurement C (-100 VDC, 30 min)
Test H

RELATIONSHIP BETWEEN

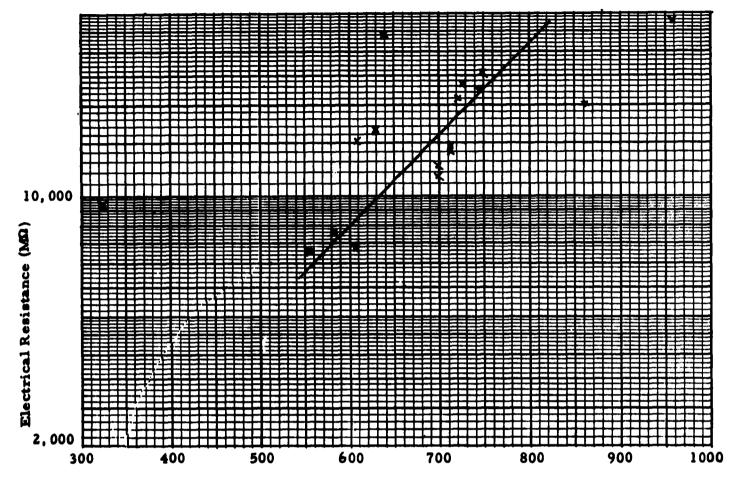
R+
DERIVED FROM MEASUREMENT C

AND

TIME TO FAILURE ON TEST H

FOR GROUP V UNITS

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)

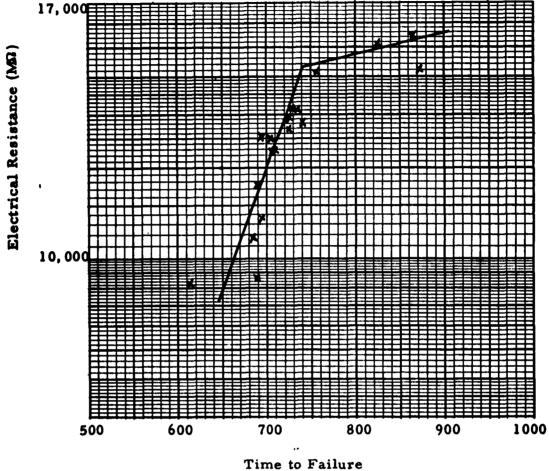


Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP I UNITS ON TEST H

(Definition of Failure: electrical resistance <100 M Ω at Test H conditions)

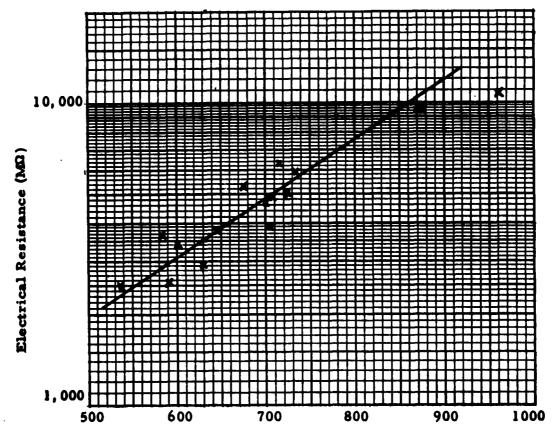
Figure 17



Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP II UNITS ON TEST H

(Definition of Failure: electrical resistance <100 MΩ at Test H conditions)

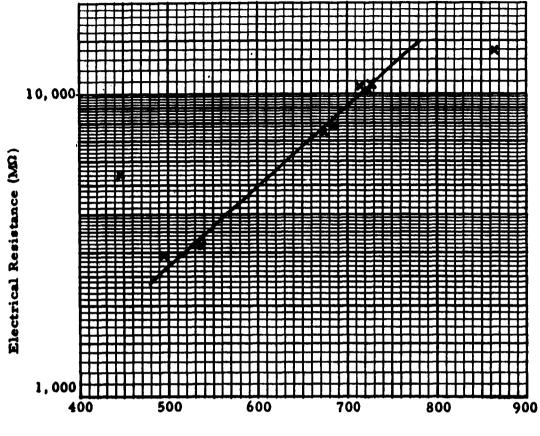


Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP III UNITS ON TEST H

(Definition of Failure: electrical resistance $< 100 \text{ M}\Omega$ at Test H conditions)

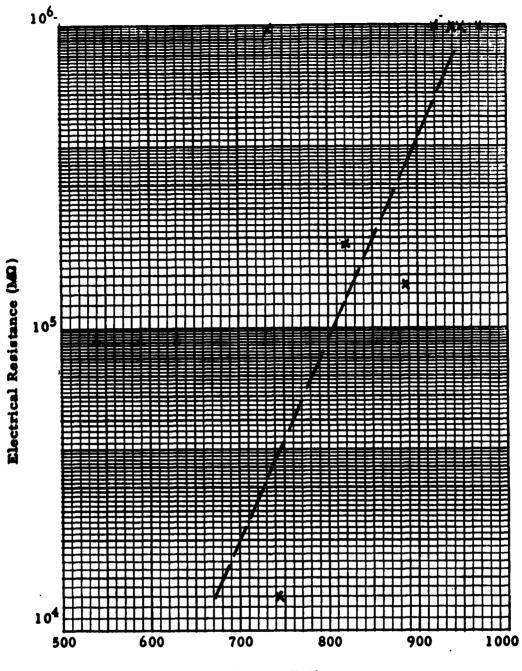
Figure 19



Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP IV UNITS ON TEST H

(Definition of Failure: electrical resistance $< 100 \ M\Omega$ at Test H conditions)

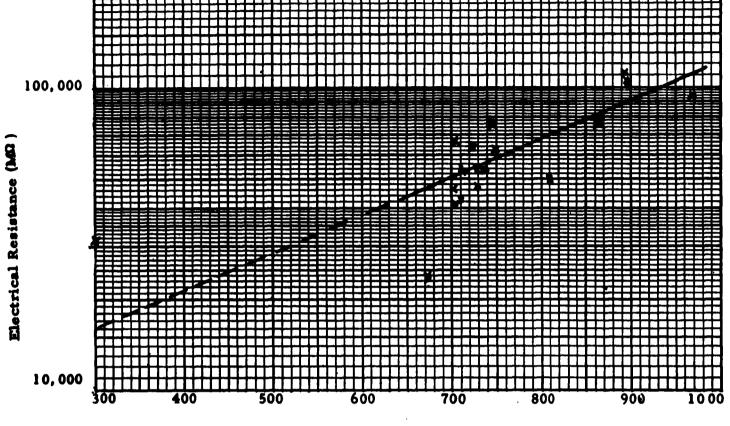


Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP IV UNITS ON TEST I

(Definition of Failure: electrical resistance < 100 M Ω at Test I conditions)

Figure 21



Time to Failure (Hours)

RELATIONSHIP BETWEEN ELECTRICAL RESISTANCE AFTER 100 HOURS AND TIME TO FAILURE FOR GROUP V UNITS ON TEST H

(Definition of Failure: electrical resistance < 100 MO at Test H conditions)

Figure 22

of life test the resistance of the capacitors drops approximately one order of magnitude at 150°C. In effect, Figures 17-22 relate resistance of the capacitors after mild degradation with time to failure. In general, it appears that there is some connection between resistance after mild degradation and time to failure, although the behavior of a few units differs with that of most of the units.

4.3 Study of Charge and Discharge Currents

Recent work at Linden Laboratories, Inc., ⁴ suggests that there is some correlation between discharge current and the stability of titania dielectrics on life test. It has been found that large discharge currents indicate unstable dielectrics.

In the process of charging a dielectric, space charges are stored in it because of inhomogeneities in the dielectric material. It is assumed that a homogeneous dielectric would be most stable on life test and that this stability would decrease as inhomogeneities are introduced. When the capacitors are discharged, the space charges produce a current in the external circuit which exists long after the current associated with the plate charge has decayed to an infinitesimally small value. The magnitude of the discharge current is therefore an indication of the inhomogeneity in the ceramic dielectric, and further, of the stability of the dielectric on life test. In other words, a capacitor exhibiting a large discharge current would have a shorter life on life test than other units exhibiting smaller discharge currents.

In an initial experiment, two sample lots of capacitors made with alkaline earth titanate and having a capacitance of approximately 1 μ f at room temperature were charged with 25 VDC for 15 min at 150°C. They were then discharged through a 1 M Ω resistor after substituting a short circuit for the power supply. This circuit is shown in Figure 23.

The units were subsequently life-tested at 150°C and 25 VDC. A unit was defined a failure when its resistance at these conditions dropped below 10 M Ω . The resistance of the units at the beginning of the life test was approximately 500 M Ω .

⁴Linden Laboratories, Inc., "Crystal Chemistry of Ceramic Dielectrics," Report No. 15, July 15, 1962, Contract No. DA-36-039-SC-78912.

CIRCUIT USED FOR MEASURING DISCHARGE CURRENTS OF SAMPLE LOTS MB126 AND MB294

Figure 23

The values of current after 2 min of discharge are presented in Table 1 for the units in each lot. A graph of time to failure vs the magnitude of discharge current after 2 min for the units of Lot MB294 is presented in Figure 24. There is a marked correlation between magnitude of discharge current and brevity of life time.

The units of Lot MB126 remained on life test for 200 hr. There were no failures. The resistance dropped approximately 4% from the initial value of about 500 $M\Omega$. It is therefore clearly evident that the capacitors from Lot MB126 are considerably more stable than the capacitors from Lot MB294. On the other hand, the values of discharge currents for each lot, while not overlapping, are sufficiently close to suggest that Lot MBl 26 should have been only slightly more stable than Lot MB294 in view of the excellent correlation between time to failure and discharge current for Lot MB294, i.e., a large discharge current indicates a relatively short time to failure. In reviewing the manufacturing history of the two lots, it was noted that while the construction and nominal compositions were almost identical, the lot numbers of raw materials used in their construction were different. The conclusion resulting from this study is that while a large discharge current may be an indication of a shortened life time for titanate capacitors, other characteristics will have to be examined in order to broaden the utility of the test.

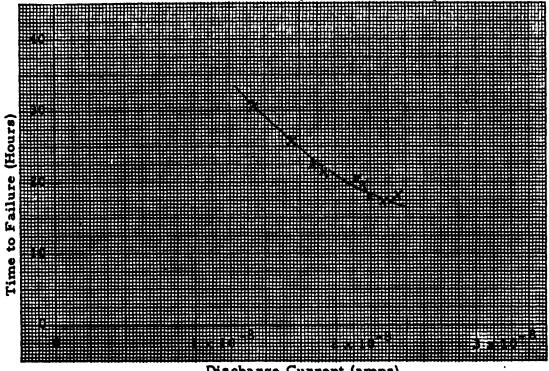
Later, the study of charge and discharge currents was expanded to include Case Size I C67 Monolythic capacitors. It was found that the instrumentation presented in Figure 23 was not sufficiently sensitive to measure accurately the currents associated with the C67 capacitor. Therefore, the circuit shown in Figure 25 was used. The Ekco 1079C instrument offers the possibility of changing the effective resistance in series with the capacitor to cover an extremely wide range of currents. The reproducibility of current measurements made with this instrument is believed to be $\pm 5\%$.

Charge and discharge curves for a C67 Monolythic capacitor at 25°C are shown in Figure 26. The capacitor was charged with 225 VDC (90 V/mil) for 10 min. At the end of this period, the power supply was replaced by a short circuit, and the capacitor was discharged externally for 1 sec through a resistance of a few thousand ohms. This was accomplished by utilizing the "set zero" setting in the Ekco 1079C electrometer. The discharge currents were then obtained using the normal ranges of the meter.

An interesting determination resulting from this study is that discharge current can be fitted to the following expression:

TABLE 1
DISCHARGE CURRENT AFTER 2 MIN

Lot MB126	Lot MB294
0.96 x 10 ⁻⁸ amp	1.83×10^{-8} amp
0.84×10^{-8} amp	2.10×10^{-8} amp
0.75×10^{-8} amp	2.34×10^{-8} amp
0.67×10^{-8} amp	2.25×10^{-8} amp
0.99×10^{-8} amp	2.40×10^{-8} amp
$0.91 \times 10^{-8} \text{ amp}$	2.16×10^{-8} amp
1.20×10^{-8} amp	1.68 x 10 ⁻⁸ amp
	2.34×10^{-8} amp
	1.92×10^{-8} amp
	1.44×10^{-8} amp



Discharge Current (amps)
(After 2 minutes)

Testing Sequence

Charged (25 VDC, 150°C, 15 min)

Discharged (through 1 MM resistor)

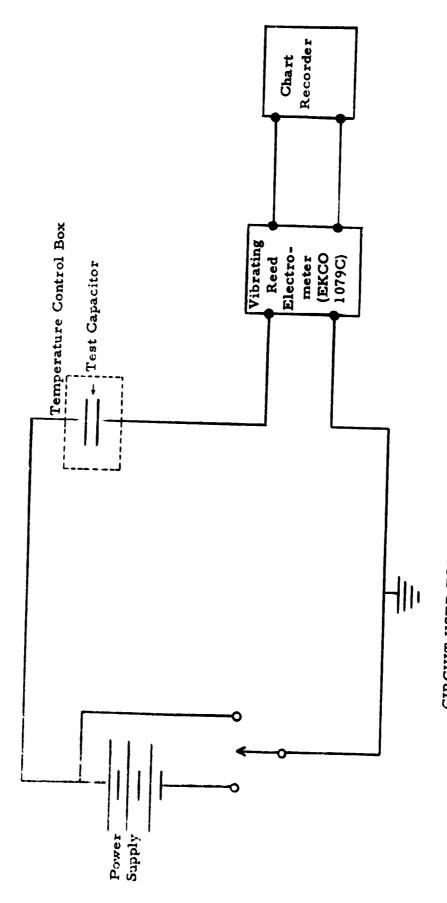
Life Test (150°C, 25 VDC)

Graph Showing

TIME TO FAILURE VS DISCHARGE CURRENT AFTER 2 MIN. FOR LOT MB294 UNITS

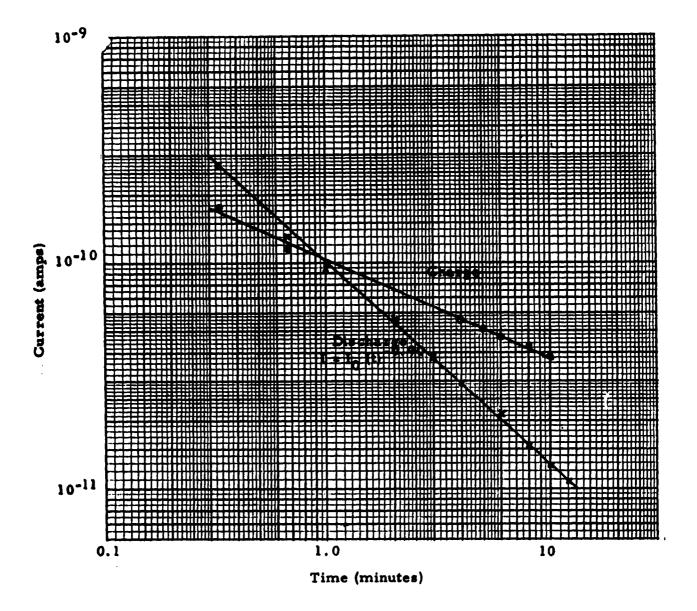
(Definition of Failure: electrical resistance $< 10 \text{ M}\Omega$ at Life Test conditions)

Figure 24



CIRCUIT USED FOR MEASURING CHARGE AND DISCHARGE CURRENT OF C67 CASE SIZE I MONOLYTHIC CAPACITORS

Figure 25



Charge Conditions: 225 VDC (90 V/mil), 25°C

Discharge Conditions: 25°C

Graph Showing

CHARGE AND DISCHARGE CURRENTS
FOR
C67 CASE SIZE I MONOLYTHIC CAPACITOR (26000 μμf)

Figure 26

$$I = I_0 t^{-n}$$

where

I = discharge current

 $I_0 = current at t = 1$

t = time

-n = slope of the curve

This formula is dissimilar to that for the current discharge of an ideal capacitor. It indicates that the source of current is not the plates of the capacitor but rather probably originates in the ceramic dielectric.

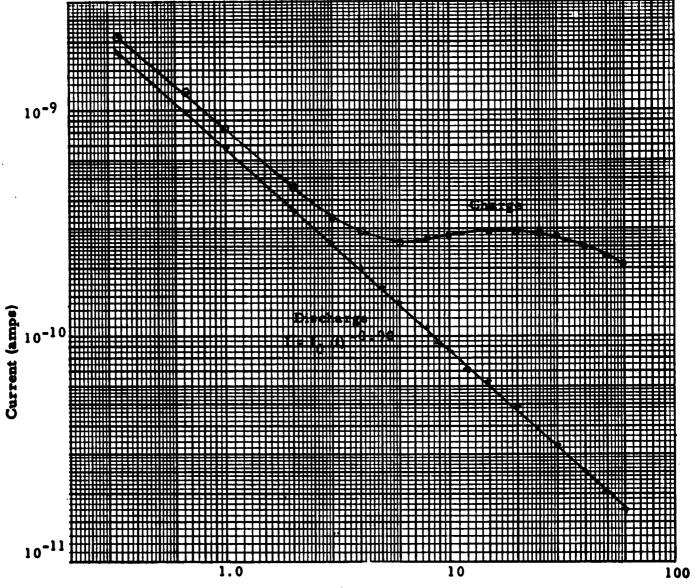
Figure 27 shows charge and discharge currents for a C67 Monolythic capacitor charged at $150\,^{\circ}$ C for 60 min with 225 VDC (90 V/mil) and then discharged for 60 min. The signs of the currents are omitted for easier comparison of charge and discharge curves. It is interesting to note that the absolute values of charge and discharge currents are similar and can be fitted to the expression $I = I_0 t^{-n}$ over much of the range.

Figure 28 presents charge and discharge currents for a C67 Monolythic capacitor which had been previously life-tested at 150°C with a voltage stress of 75 V/mil for 1600 hr. In the course of the life test, the resistance of the unit had decreased approximately four orders of magnitude.

The degraded capacitor was charged at 100°C with 225 VDC for 60 min and then discharged. In this case, the absolute values of charge and discharge current at any time are orders of magnitude apart. In order that the reader may make a comparison, the charge and discharge currents at 100°C for a nondegraded unit are presented in Figure 29. It can be seen that the discharge current for a degraded unit is approximately one order of magnitude greater than that for a nondegraded unit. The sustained application of a large DC stress apparently produces a situation which encourages the trapping of charges during charging.

4.4 Matrix I Testing

As reported in the First Quarterly Report, the beginning of Matrix I testing has been delayed until sufficient information is accumulated from the pre-Matrix I testing so that the Matrix I conditions can be optimized. This is in accordance with verbal agreement between the contractor's technical representative and the Sprague Electric Company.



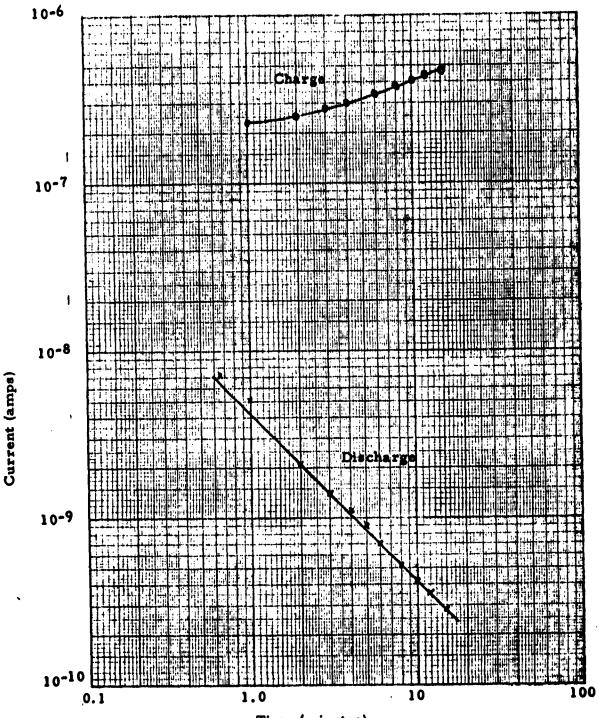
Time (minutes)

Charge Conditions: 225 VDC (90 V/mil), 150°C

Discharge Conditions: 150°C

Graph Showing

CHARGE AND DISCHARGE CURRENTS
FOR
C67 CASE SIZE I MONOLYTHIC CAPACITOR (26000 μμf)

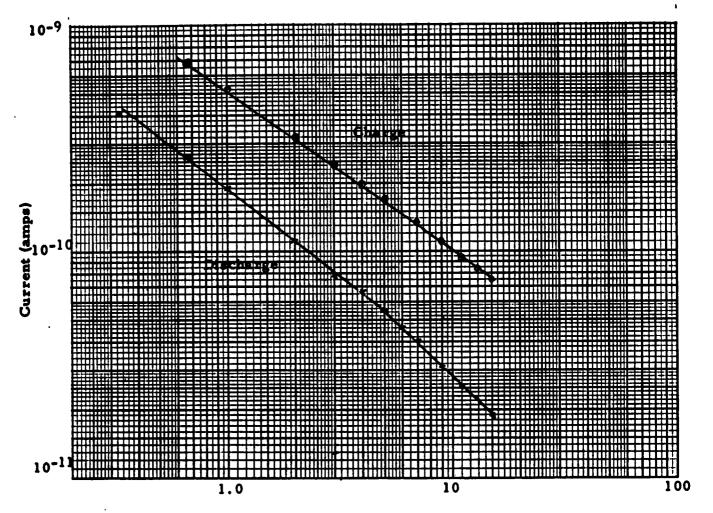


Time (minutes)

Charge Conditions: 225 VDC (90 V/mil), 100°C Discharge Conditions: 100°C

Graph Showing

CHARGE AND DISCHARGE CURRENTS FOR LIFE-TESTED C67 CASE SIZE I MONOLYTHIC CAPACITORS



Time (minutes)

Charge Conditions: 225 VDC (90 V/mil), 100°C

Discharge Conditions: 100°C

Graph Showing

CHARGE AND DISCHARGE CURRENTS
FOR
C67 CASE SIZE I MONOLYTHIC CAPACITOR (≅6000 μμf)

Figure 29

SECTION 5

CONCLUSIONS

- (1) Application of the three AC voltages experimented with in the pre-matrix testing does not offer a way to eliminate early failures. The ionic nature of titanate wearout appears to preclude any effective use of AC voltage as aging devices.
- (2) There appears to be a correlation between electrical resistance at some time during DC voltage application and time to failure. The results of accelerated life testing indicate that a DC aging process can reduce lifetime, but it may also eliminate many early failures.
- (3) No final conclusions can be made on the efficacy of using discharge currents as indicators of life performance.

SECTION 6

PROGRAM FOR NEXT QUARTER

A study will be made of charge and discharge curves from Case Size I C67 Monolythic capacitors in order to determine their possible use as indicators of life performance.

SECTION 7

IDENTIFICATION OF PERSONNEL

Personnel	Hours
J. Dziok W. Estes J. Fabricius H. Geller E. Jamros	1.0 146.1 20.0 67.6 118.0
E. Jones M. Malanga G. Olsen T. Prokopowicz D. Reid	1.5 5.0 1.0 50.0 44.0
F. Schoenfeld W. Tatem R. Trottier K. Whitney J. Willey	15.0 219.0 60.0 6.0 102.6
Total	856.7

SECTION 8

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